

Chemistry 146A
Advanced Laboratory in Organic Chemistry
Fall 2016

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This manual is the collaborative effort of the organic chemistry faculty Caitlin Binder, Rebecca Braslau, and Daniel Palleros with the invaluable help of Chris Murphy and Chris Bailey.

CHEM 146A – Advanced Organic Chemistry Lab
UCSC, Department of Chemistry and Biochemistry

Instructor: Dr. Caitlin Binder
Email: cambinde@ucsc.edu

Office Location: Thimann Labs 313
Office hours: Fridays 2 – 4 p.m.

Teaching Assistants: Chad Higa (cmhiga), Rachel Snelling (rsnellin), & Jen Petraitis (jpetrait)

Course Prerequisites: CHEM 108L/M, 110L **Course Fees:** \$55 materials fee

Lab Meetings: 01A-TuTh 1:30 – 5:30 p.m.; 01B-MW 12 – 4 p.m., see schedule

Required Materials

- Any Experimental Organic Chemistry textbook, such as...
 - Mohrig, J. R., *et. al* "Techniques in Organic Chemistry" Freeman (any edition)
 - Palleros, D. R. "Experimental Organic Chemistry" Wiley, 2000.
- Lab Notebook – no carbon copies needed, get a fresh new notebook of any kind
- Goggles & Lab Coat (provided) or you may buy your own approved safety goggles.
- Access to course website for supplemental materials and updates (URL below)

Required Assignments

There are four distinct laboratory experiments to be completed and there will be four written assignments to be done in a professional, complete manner. More details will be made available in class. You must also complete a safety orientation activity at the beginning of the class (see below). The minimum passing grade for each experiment is 70%.

Safety Orientation

Students will self-conduct a tour of the lab space to locate the safety features of the labs. A brief activity will be completed and shown to the instructor before leaving lab on the first day.

Laboratory Notebook

An up-to-date, current laboratory notebook should be kept while doing experiments. The notebook will be checked periodically without notice, and will be turned in with the reports (for more info about keeping a lab notebook, see below).

Absences

If you absolutely need to miss a lab, *pre-arrange your absence with the instructor.*

Experiment 1 and 2 **cannot** be postponed, however there is some flexibility in Experiments 3 & 4. This does not mean that you are free to miss labs on a causal basis: email the instructor ahead of your missed class. **Non-prearranged absences can be grounds for a Fail (F) in this class. No make-up laboratory sessions will be arranged!**

Illness. Casts and Crutches. Pregnancy

If you are ill, you **should not attend** lab and, as stated above, should contact the instructor as soon as possible. In the circumstance that you must wear a cast, a sling or use crutches, please contact the instructor immediately, before your next lab section, so we can arrange the best possible accommodations for you. Also contact the DRC (Disability Resource Center 831-459-2089) as soon as possible. If you are pregnant or intend to get pregnant during the course of this quarter, please contact the instructor before your next lab section to obtain important information about chemicals and pregnancy. Also, we recommend that you consult with your physician about this subject.

Web site

<https://acrochem.sites.ucsc.edu/chem-146a/>

Assignments Overview

****All drafts & reports must be printed and in-hand at the beginning of lab****

Experiment 1: Acid-Base Extraction & Column Chromatography

In the first two lab periods, students will repeat 2 labs from the 108 series: acid-base extraction of Excedrin components and separation of an essential oil using column chromatography. Before the experiment, students type a short paragraph describing the theory behind each experiment and prepare the lab notebook with the concise experimental details. The main results are typed in another brief paragraph, tying the results back to the theory behind the experiment.

First half due on October 3-4; Second half due on October 5-6.

Experiment 2: Identification of a Single Unknown

The identification of an unknown compound with no more than nine carbon atoms by spectroscopy, chemicals tests, and provided $^1\text{H-NMR}$.

First draft due on October 17-18; Revised Final Report due in class October 24-25.

Experiment 3: Separation & Identification of Two Unknowns

Each student will be given a unique mixture of two organic unknowns, which will be separated by standard laboratory procedures. After purification, each of the clean compounds will then be subjected to a variety of spectroscopic analyses (IR, ^1H and ^{13}C NMR) outlined in class. The student should use this information to successfully identify each of them. The lab report will consist of a record of the separation and purification schemes and yields obtained for each step, the fully assigned and interpreted spectra, and a discussion of the structural analysis.

First draft due November 7-8; Revised Final Report due in class November 14-15.

Experiment 4: Synthetic Methodology

One of two projects will be completed, as described below.

Progress report (results & experimentals) due November 21-22. Draft due on the last lab meeting (11/30-12/1). Revised Final Report due in your TA's mailbox Monday, December 5th by 5 p.m.

Experiment 4A: CDI Coupling of Unknowns

Each student in Group A will be given a set of two unknowns to react in a CDI-mediated amide coupling. Students will carry out the reaction twice to maximize yield, elucidate the structures, then repeat with two additional unknowns.

Experiment 4B: Optimization of the Synthesis of Phenacetin from Tylenol

Group B will collaborate on the optimization of the historically low-yielding transformation of acetaminophen into phenacetin. A list of variables and alternate reagents is provided and students will determine which variables will be changed by the end of the second week of lab. The lab report will combine the results for the entire section with critical analysis and determination of the optimal conditions based on yield and purity of products.

Grading

Lab Orientation, 1%

Participation & Preparedness: 5%

Experiment 1: 90 points; 9%

Experiment 2: 20%

Experiment 3: 25%

Experiment 4: 20%

Technical Skill / Lab Notebook / Safety Compliance (evaluated by the instructor and TA): 20%

Note: all experiments and written assignments must be completed on time to pass the course. A minimum grade of 70% will be needed to pass each experiment. Grades may be curved based on class performance. A = 90-100%; B = 80-89%; C = 70-79%; D = 60-69%; F = < 50%.

Introduction to Chemistry 146A

Above all else, safety will be emphasized. Please read the Laboratory Safety section carefully. You will be tested on it at the beginning of the course. You can find a safety quiz to test your knowledge on line, at the class web site. Students who do not come properly dressed for the lab will not be allowed in. This also includes the first day of class or check-in, which may result in you being dropped from the class. Students who do not follow the safety rules during the course of the lab will be asked to leave. This may result in failing the class.

We want to emphasize to you that a collegial and professional atmosphere is mandatory in these lab sections. The teaching assistant and instructor are here to help you. Our philosophy is that we are here to act as your research advisors and, hopefully, to convey to you some of our own passion for organic chemistry. **Your part of the deal is thinking, preparing and asking questions, no matter how stupid you might think they are.** Collegial interaction and cooperation is encouraged. That means that you should feel free to consult with each other and with the TA about the chemistry that you are doing. It doesn't mean that the TA or another student does your lab work for you.

Please remember that the amount of time available for laboratory work is limited. The laboratory is the place to make observations; use time outside the lab for planning and consulting the literature. It is essential that you have a plan of action when you arrive at the laboratory. This means that you must plan ahead for the experiments. You can't afford to look information up as you need it.

The accurate and concise communication of research results and the ideas generated by these results is paramount in every branch of science. Maintenance of a neat, professional, and **permanent laboratory notebook** is an important objective of this course. Whether you write large or small, neat or not so, if the information in your notebook cannot be read, then that information is worthless. In addition, please remember that "bad results" are at least as important as "good results." All entries in your notebook should be made in ball point pen (so it will not "run" if wet). Your lab book must have bound, consecutively numbered pages.

Take good care of your bench space. Keep it clean and tidy at all times. Keep the reagents counter and the balances clean. If you spill a small amount of a solid when transferring or weighing it, clean it with wet paper towels and dispose of the towels in the solid-waste container; wear gloves while doing it. Keep reagent bottles and Erlenmeyer flasks well capped. Do not move reagents around.

All products made or purified in the lab should be stored in a screw-cap vial or a plastic bag that is properly labeled with the compound's name, melting point (if applicable), mass, your name and the date. The vials or bags are to be turned in immediately after completion of the experiment to the stockroom or your TA unless requested to do otherwise.

Have fun, and learn as much as you can! Again, your TA and instructor are committed and approachable!

LAB SCHEDULE

Arrive on time and dress appropriately for every lab meeting, even if no experiment will be performed that day. Bring the lab manual, lab notebook, and textbook to every lab.

	Monday/Tuesday	Wednesday/Thursday
1	9/26-9/27 Introduction, Orientation, Safety ** Students must be present this day to secure their space **	9/28-9/29 Experiment 1: Group A - Columns Group B - Acid-Base Extraction
2	10/3-10/4 - Experiment 1: Group B - Columns Group A - Acid-Base Extraction Exp 1 report (1st half) due	10/5-10/6 – Lab Clean-up/transition Exp 1 report (2nd half) due NMR Tours & Worksheet Experiment 2 preparation
3	10/10-10/11 Experiment 2: Identification of an Unknown	10/12-10/13 Experiment 2: Identification of an Unknown
4	10/17-10/18 – Lab Clean-up/transition Experiment 2 draft due Writing workshop, laptops ok Experiment 3 preparation Group A – ChemDraw & SciFinder, go to DSC after writing workshop (time TBD)	10/19-10/20 Experiment 3, Group A Separation & Identification of Two Unknowns (Extraction) Group B – ChemDraw & SciFinder @ beginning of lab time, no lab after
Chemdraw & SciFinder tutorials in McHenry Library Digital Scholarship Commons. Register for passwords & find the DSC by 10/14. Space reserved – pay attention to announcements in lab for time assignment.		
5	10/24-10/25 - Experiment 3, Group B Separation & Identification of Two Unknowns (Extraction) Experiment 2 Report Due Group A – no lab, except to turn in report	10/26-10/27 – Experiment 3: Separation & Identification of Two Unknowns (Extraction)
6	10/31-11/1 – Experiment 3: Separation & Identification of Two Unknowns (Purification)	11/2-11/3 – Experiment 3: Separation & Identification of Two Unknowns (Characterization)
7	11/7-11/8 – Experiment 3: loose ends Lab clean-up/transition Experiment 3 - 1st draft due Writing workshop – peer review	11/9-11/10 – Experiment 4 Group A: CDI Coupling of Unknowns Group B – no lab, encouraged to come during lab for help with report
8	11/14-11/15 – Experiment 4 Group B: Optimization of Phenacetin Group A – no lab, encouraged to come to lab for help with report	11/16-11/17 – Experiment 4 (both groups) Experiment 3 Report Due
9	11/21-11/22 – Experiment 4 - both Exp 4 Progress Report Due (Results & Experimentals)	11/23-11/24 – Thanksgiving – no lab
10	11/28-11/29 – Experiment 4 - both	11/30-12/1 – Dry Lab Experiment 4 Draft Due Writing workshop – peer review Lab Clean-up